201-14884

HIGH PRODUCTION VOLUME (HPV) CHALLENGE PROGRAM

TEST PLAN
FOR

ETHYL(3-METHYLPHENYL)-AMINO ACETONITRILE

(CAS NO.: 63133-74-4)

PREPARED BY:

EASTMAN CHEMICAL COMPANY

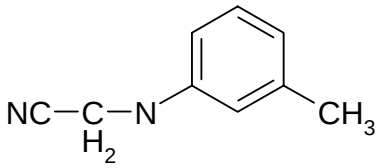
RECEIVED
OPPT CBIC
03 DEC 10 AM 10: 18

December 8, 2003

OVERVIEW

The Eastman Chemical Company hereby submit for review and public comment the test plan for ethyl(3-methylphenyl)-amino acetonitrile (EMAA; CAS NO.: 63133-74-4) under the Environmental Protection Agency's (EPA) High Production Volume (HPV) Chemical Challenge Program. It is the intent of our company to use existing data on EMAA in conjunction with EPA-acceptable predictive computer models to adequately fulfill the Screening Information Data Set (SIDS) for the physicochemical, environmental fate, ecotoxicity test, and human health effects endpoints. In addition, we believe that due to the sole use of this material as an industrial intermediate used solely on-site in a closed-system manufacturing process a reduced set of data are needed. In total the data submitted are believed to be adequate to fulfill all the requirements of the HPV program without need for the conduct any new or additional tests.

TEST PLAN SUMMARY

CAS No. 63133-74-4 	Information	OECD Study	Other	Estimation	GLP	Acceptable	New Testing Required
STUDY	Y/N	Y/N	Y/N	Y/N	Y/N	Y/N	Y/N
PHYSICAL-CHEMICAL DATA							
Melting Point	Y	-	Y	-	N	Y	N
Boiling Point	Y	-	Y	-	N	Y	N
Vapor Pressure	Y	-	-	Y	N	Y	N
Partition Coefficient	Y	-	-	Y	N	Y	N
Water Solubility	Y	-	-	Y	N	Y	N
ENVIRONMENTAL FATE ENDPOINTS							
Photodegradation	Y	-	-	Y	N	Y	N
Stability in Water	Y	-	-	Y	-	Y	N
Biodegradation	Y	Y	-	-	Y	Y	N
Transport between Environmental Compartments (Fugacity)	Y	-	-	Y	N	Y	N
ECOTOXICITY							
Acute Toxicity to Fish	Y	Y	-	-	Y	Y	N
Acute Toxicity to Aquatic Invertebrates	Y	Y	-	-	Y	Y	N
Toxicity to Aquatic Plants	Y	Y	-	-	Y	Y	N
TOXICOLOGICAL DATA							
Acute Toxicity	Y	N	Y	-	N	Y	N
Repeated Dose Toxicity	N	-	-	-	-	-	N
Genetic Toxicity – Mutation	Y	Y	-	-	Y	Y	N
Genetic Toxicity – Chromosomal Aberrations	Y	Y	-	-	Y	Y	N
Developmental Toxicity	N	-	-	-	-	-	Y
Toxicity to Reproduction	N	-	-	-	-	-	N

TEST PLAN DESCRIPTION FOR EACH SIDS ENDPOINT

A. Physicochemical

Melting point - Data for this endpoint were obtained using a measured value.

Boiling Point - Data for this endpoint were obtained using a measured value.

Vapor Pressure - A value for this endpoint was obtained using a computer estimation-modeling program within EPIWIN(1).

Partition Coefficient - A value for this endpoint was obtained using a computer estimation-modeling program within EPIWIN.

Water Solubility - A value for this endpoint was obtained using a computer estimation-modeling program within EPIWIN.

Conclusion: All end points have been satisfied by utilizing data obtained from the various physical chemical data modeling programs within EPIWIN or from use of measured data. Estimation models within EPIWIN have been noted by the Agency as acceptable in lieu of actual data or values identified from textbooks. No new testing is required.

B. Environmental Fate

Photodegradation - A value for this endpoint was obtained using AOPWIN, a computer estimation-modeling program within EPIWIN (1).

Stability in Water - No data are available. A technical discussion describing the stability of EMAA in water has been provided.

Biodegradation - This endpoint was satisfied through the use of a study that followed OECD-301B guidelines and was conducted under GLP assurances.

Fugacity - A value for this endpoint was obtained using the EQC Level III partitioning computer estimation model within EPIWIN.

Conclusion: Except water stability all endpoints have been filled with data utilizing acceptable methodologies and of sufficient quality to fulfill these endpoints. No new testing is required.

C. Ecotoxicity Data

Acute Toxicity to Fish - This endpoint is filled by data from a study that followed OECD TG-203 and was conducted under GLP assurances.

Acute Toxicity to Aquatic Invertebrates - This endpoint is filled by data from a study that followed OECD TG-202 and was conducted under GLP assurances.

Toxicity to Aquatic Plants - This endpoint is filled by data from a study that followed OECD TG-201 and was conducted under GLP assurances.

Conclusion: All endpoints, but algal toxicity, have been satisfied with data from studies that were conducted using established OECD guidelines and GLP assurances. No new testing is required.

D. Toxicological Data

Acute Toxicity - This endpoint is filled by data from studies conducted in rats and mice that assessed the toxicity of EMAA following oral exposure and in guinea pigs subsequent to a dermal exposure. Although the studies did not follow standardized guideline protocols they were deemed as "reliable with restrictions".

Repeat Dose Toxicity - No data are available other than that which is contained in the OECD 421 developmental and reproductive toxicity screening study. However, the sole use of this material is as an industrial intermediate and arguments are presented to support a reduced set of testing needs that excludes repeat dose toxicity studies.

Genetic Toxicity

Mutation - This endpoint is filled with a study that followed OECD guideline 471 and was conducted under GLP assurances. The quality of this study was deemed as "reliable without restrictions".

Aberration - This endpoint is filled with data from an *in vitro* study using Chinese hamster ovary (CHO) cells that followed OECD guideline 473 and was conducted under GLP assurances. The quality of this study was deemed as "reliable without restrictions".

Developmental

Toxicity - No data are available. This endpoint is to be filled by data from an oral exposure study in rats that will follow OECD guideline 421 and will be conducted under GLP assurances. This protocol evaluates both developmental and reproductive toxicity potential.

Reproductive

Toxicity - No data are available, nor are data needed due to the nature of the manufacture and use of this compound. Nevertheless, this endpoint is to be filled by data from an oral exposure study in rats that will follow OECD guideline 421 and will be conducted under GLP assurances. This protocol evaluates both developmental and reproductive toxicity potential.

Conclusion: All endpoints have been satisfied with data from studies whose methods followed established OECD guidelines and GLP assurances. The only data needed to be obtained are those assessing developmental toxicity potential.

SIDS DATA SUMMARY

Data assessing the various physicochemical properties (melting point, boiling point, vapor pressure, partition coefficient, and water solubility) for EMAA were obtained from estimations using the models within EPIWIN or from measured values. These data indicate that EMAA is a liquid at room temperature (MP <0 ^{o}C) with a low vapor pressure (0.027 hPa at 25 °C) and a boiling point in excess of 250 °C. It has an octanol to water partition coefficient (K_{ow}) of 2.73 and an estimated water solubility of 252 mg/L.

The assessment of the environmental fate endpoints photodegradation and biodegradation indicate the material is capable of being degraded by photochemical reactions but appears to not be readily degraded using biological processes. Fugacity predictions indicate a very limited amount of partitioning into the air, with 99% estimated to be distributed into the soil (68.2%) and water (31.4%). It is estimated that the material is hydrolytically stable. However, since this material is manufactured in closed and is not transported off-site environmental exposures are not likely to occur.

The data from the various studies conducted to assess ecotoxicity potential indicate EMAA may be toxic to fish, daphnia, and algae. The LC_{50}/EC_{50} concentrations for the respective species are 27.7, 40, and <3 mg/L.

The acute toxicity of EMAA following oral exposure is moderate with an LD_{50} value of approximately 200 - 400 mg/kg in arts and 400 – 800 mg/kg in mice. Material was not toxic following dermal exposure. The material is not genotoxic based on the negative results of an Ames study and an *in vitro* chromosomal aberration study. Data from repeated exposure studies are not deemed necessary due to the use of this material as an intermediate and be the manner in which it is manufactured and handled. Data are needed to assess the developmental toxicity potential. While data are not needed for assessing reproductive toxicity, results of a study following OECD guideline 421 will provide information to adequately assess this endpoint.

In conclusion, data to adequately assess all the SIDS endpoints are currently available or will be available. Importantly, due to its only known use as a closed system on-site industrial intermediate with no known direct applications within consumer products, exposure to the general public is not anticipated and exposure to workers is managed through prudent industrial hygiene practices.

JUSTIFICATION TO SUPPORT REDUCED TESTING

It is believed that a reduced set of hazard data are needed for EMAA due to the fact that this compound is a closed-system industrial intermediate used only on-site at one manufacturing facility and is not transported. The documentation for the basis of this claim is detailed in the attached appendix.

EVALUATION OF DATA FOR QUALITY AND ACCEPTABILITY

The collected data were reviewed for quality and acceptability following the general US EPA guidance (2) and the systematic approach described by Klimisch *et al.* (3). These methods include consideration of the reliability, relevance and adequacy of the data in evaluating their usefulness for hazard assessment purposes. The codification described by Klimisch specifies four categories of reliability for describing data adequacy. These are:

1. Reliable without Restriction: Includes studies or data complying with Good Laboratory Practice (GLP) procedures, or with valid and/or internationally accepted testing guidelines, or in which the test parameters are documented and comparable to these guidelines.

2. Reliable with Restrictions: Includes studies or data in which test parameters are documented but vary slightly from testing guidelines.

3. Not Reliable: Includes studies or data in which there are interferences, or that use non-relevant organisms or exposure routes, or which were carried out using unacceptable methods, or where documentation is insufficient.

4. Not Assignable: Includes studies or data in which insufficient detail is reported to assign a rating, e.g., listed in abstracts or secondary literature.

REFERENCES

1. EPIWIN, Version 3.11, Syracuse Research Corporation, Syracuse, New York.

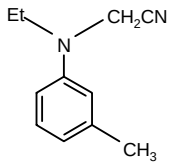
2. USEPA (1998). 3.4 Guidance for Meeting the SIDS Requirements (The SIDS Guide). Guidance for the HPV Challenge Program. Dated 11/2/98.

3. Klimisch, H.-J., Andreae, M., and Tillmann, U. (1997). A Systematic Approach for Evaluating the Quality of Experimental Toxicological and Ecotoxicological Data. *Regul. Toxicol. Pharmacol.* 25:1-5.

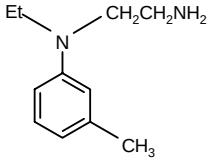
Appendix I

Reduced Testing Claims for PM 1095 and PM 1096
EPA HPV Challenge Program

The following information is presented to support exemption claims for reduced SIDS testing under the EPA HPV Challenge Program for ethyl (3-methylphenyl)-amino acetonitrile (CAS No. 063133-74-4, hereinafter referred to as EMAA) and N-ethyl-N-(3-methylphenyl)-1,2-ethanediamine (CAS No. 019248-13-6, hereinafter referred to as EMPE).



EMAA



EMPE

The information is presented for both chemicals in this document, since the former (EMAA) is converted to the latter (EMPE) in the overall manufacturing scheme to produce the photographic color developer CD-3 [4-(N-ethyl-N-2-methanesulfonylaminoethyl)-2-methylphenylenediamine sesquisulfate monohydrate, CAS No. 025646-71-3]. The overall chemistry and manufacturing sequences are presented in Attachment I.

The overall organization and content of the information are consistent with the requirements and criteria prescribed in the "SIDS Manual: Screening Information Data Set Manual of the OECD Program on the Cooperative Investigation of High Production Volume Chemicals", July 1997. Both EMAA and EMPE are intermediates which are manufactured and consumed for the sole purpose of the manufacture of color developer CD-3. Both are Type (a) closed system isolated intermediates which are stored in controlled on-site facilities and subsequently fully consumed in the same on-site facilities.

I. Information on Sites

A. Number of Sites

EMAA and EMPE are manufactured and consumed at a single site, Tennessee Eastman Division of Eastman Chemical Company, in Kingsport, Tennessee.

B. Basis for "Closed Process" Conclusion

A process flow diagram covering major unit operations at all steps in the CD-3 manufacturing process is provided in Attachment I. All points of potential release and waste streams are indicated on the process flow diagram. Each point of

potential release and each waste stream are discussed below, with reference to these specific points from the process flow sheet.

The potential points of release may be generally categorized as 1) aqueous wastes, 2) organic wastes, 3) process vessel vents, and 4) sampling. All aqueous waste streams are directed to an on-site wastewater treatment facility, fully qualified and registered as a hazardous waste disposal facility. All organic waste streams are directed to an on-site incinerator facility, also fully qualified and registered as a hazardous waste disposal facility. The data for the two substances of interest in aqueous waste or organic waste streams are taken from internal detailed waste stream documentation, based on either measurement data or estimation based upon profound process knowledge. Monitoring data are limited for the potential release of the materials of interest via process vessel vents. However, detailed ASPEN modeling data generated in accordance with Title V Vent permit requirements are provided in the discussion below for each process vessel vent in the process flow diagram. The details of each process sampling point are also included in the discussion below. Unused portions of samples are destroyed by incineration in an on-site, hazardous waste disposal facility.

All process steps described below are controlled via a GSE D3 Distributed Control System utilizing FlexBatch sequence programming software.

The paragraph numbers in the following narrative references the process flow diagram in Attachment I.

1. EMAA Preparation - Reactants are mixed in one of two EMAA reactors in parallel to produce a crude grade of approximately 90% EMAA and 10% starting materials and by-products. The EMAA is exposed to a maximum temperature of 65ºC at atmospheric pressure. Each reactor is vented to the atmosphere via roof vents. A water decant is incorporated at the final stages of the process to separate some water soluble inorganic and organic by-products from the crude EMAA upper layer which remains in the reactor. The crude EMAA is sampled for analysis at the end of the process. The crude EMAA is transferred directly to a vacuum still for purification by distillation.

1a. EMAA Preparation Process Vents – Each of the two EMAA Reactors in parallel and all ancillary equipment are vented to the atmosphere via roof vents. Detailed Aspen modeling for Title V Vent Permits indicates no EMAA emission from vents under the most severe process conditions of temperature and pressure.

1b. EMAA Preparation Aqueous Decant – The single aqueous decant is ultimately directed to the on-site Industrial Waste Water Treatment Facility (WWT). The aqueous decant from the EMAA reactors is initially directed to a secondary decanter through conductivity probes to detect the EMAA interface.

The aqueous layer from the secondary decanter, also equipped with conductivity probes to detect the EMAA interface, directs the aqueous layer to an aqueous layer treatment tank where it undergoes a chemical treatment and is finally discharged via pump out receiver to WWT. Any EMAA collected in secondary decanter is recycled directly back to the EMAA reactor. Detailed waste stream documentation indicates that approximately 12 pounds per batch (~12,000 lbs./yr.) of EMAA are discharged to the WWT via the decant steps.

1s. EMAA Preparation Sampling – A 4 oz. dip sample is removed through the loading port of the EMAA reactor with the contents at approximately 60ºC and atmospheric pressure. The operator wears safety glasses and a face shield or safety goggles, rubber gloves, a plastic apron over flame retardant coveralls, and static dissipating leather shoes.

2. EMAA Distillation – Three parallel EMAA batch stills with packed columns separate EMAA from low boilers and unconsumed reactants via vacuum distillation. The product cuts, containing EMAA at 98% nominal assay, is directed to a distilled EMAA storage tank.

2a. EMAA Distillation Process Vents – Each batch still vents via condensers to 2-stage steam jet system. Steam condensate from steam jet after condenser collects in a seal pot that overflows to WWT. Still residue (high boiling tar) is discharged via a tar dilution tank to a hazardous waste dumpster for incineration. The tar dilution tank, condenser, and receiver vent to atmosphere via a roof vent. A first cut receiver is discharged to an indoor First Cuts storage tank for recycle to normal production. The First Cuts storage tank is vented to atmosphere via a roof vent and is maintained at ambient temperature. Product receivers are transferred via an indoor measuring tank to an outside storage tank. The measuring tank is vented to atmosphere via a roof vent. Detailed Aspen modeling for Title V Vent Permits indicates no EMAA emissions from the batch stills and associated vents.

2b. EMAA Distillation Organic Wastes (Tars) – Once per three to four distillation batches, tar residue in the batch still is discharged via a tar dilution tank to a hazardous waste disposal facility. Detailed waste stream documentation indicates that no EMAA is discharged to the hazardous waste disposal facility.

2s. EMAA Distillation Sampling – A 4 oz. sample is collected from a sample valve in the product distillate receiver. The operator wears safety glasses and a face shield or safety goggles, rubber gloves, a plastic apron over flame retardant coveralls, and static dissipating leather shoes.

3. EMAA In-Process Storage – Distilled EMAA is transferred to an outside storage tank where it is diluted with isopropyl alcohol and stored at ambient temperature and atmospheric pressure. It is pumped from the storage tank to an autoclave for conversion to EMPE.

3a. EMAA IN-Process Storage Vessel Vent – The EMAA storage tank maintains a slight nitrogen blanket and is vented to atmosphere through a conservation vent set to relieve pressure at 1 oz. Detailed Aspen modeling for Title V Vent Permits indicates no EMAA emissions from associated vents.

4. EMPE Preparation – EMAA in isopropyl alcohol solution is converted to EMPE by catalytic hydrogenation at 500 psig hydrogen pressure over a noble metal catalyst at a maximum temperature of 125^{o}C. The autoclave is cooled to 55-60^{o}C and vented. The EMPE solution in isopropyl alcohol is clarified through a filter to remove catalyst, sampled, and pumped to a crude EMPE storage tank. The recovered catalyst is washed with water, and drummed for catalyst reclamation.

Each EMPE crude batch is analyzed by gas chromatography for EMPE assay and residual EMAA, and the data are captured in a manufacturing information system. The EMPE assay averages 94% on a solvent-free basis. EMAA is virtually consumed quantitatively in the reaction, with the residual EMAA content averaging 0.01% on a solvent-free basis. The detection limit for EMAA in the analysis is estimated to be approximately 200 ppm. There is an occasional batch with an elevated level of residual EMAA in crude EMPE, generally in the 0.1% to 0.2% range,

4a. EMPE Preparation Process Venting – EMAA is transferred from the bulk storage tank at ambient temperature through a measuring tank vented to atmosphere and into the autoclave for batch wise hydrogenation. Following the reduction to crude EMPE, the autoclave is vented through a water scrubber to atmosphere. Detailed vent calculations performed with an in-house program (E.A.S.I.) for Title V Vent Permit application indicates no EPME emissions from the autoclave and associated vents.

4b. EMPE Preparation Recovered Catalyst Wash – Each batch of crude EMPE is transferred from the autoclave through a filtration system to remove any entrained catalyst. Following the batch transfer through the filters, the lines and filtration equipment are water rinsed to the interceptor sewer to remove residual alcohol, and dirty filter bags are collected and new ones installed. Approximately every 30 batches, a cartridge filter element is replaced as well. Detailed waste stream documentation indicates that on average, approximately 4 pounds of EMPE per batch are discharged to the hazardous waste disposal facility during this filtration equipment rinsing step.

4c. EMPE Preparation Catalyst Reclamation – After approximately 125 EMPE preparation batches, the catalyst is transferred from the autoclave and collected in the filtration system for reclamation. The catalyst is rinsed with water to the interceptor sewer and then filter bags and the filter cartridge are removed and placed in drums for shipment as a hazardous waste. Detailed waste stream documentation indicates that on average, approximately 4 pounds of EMPE per

batch are contained in the catalyst filters and shipped to the catalyst reclamation vendor for disposal.

4s. EMPE Preparation Sampling - A 4 oz. sample is collected from each EMPE batch as it is discharged from the autoclave. The operator wears safety glasses and a face shield or safety goggles, rubber gloves, a plastic apron over flame retardant coveralls, and static dissipating leather shoes.

5. Crude EMPE In-Process Storage – Crude EMPE in isopropyl alcohol is stored at ambient temperature in an outside storage tank. The crude EMPE is then transferred to an indoor lot tank maintained at ambient temperature and atmospheric pressure. It is pumped from the storage tank to a batch still for removal of the isopropyl alcohol by distillation.

5a. Crude EMPE In-Process Storage Vessel Vents – The outside storage tank is vented to the atmosphere through a conservation vent set to relieve pressure at 15" WC. The indoor lot tank is vented to the atmosphere via a roof vent. Detailed Aspen modeling for Title V Vent Permits indicates no EMPE emissions from the tanks and associated vents.

6. Crude EMPE Stripping (Solvent Removal) – Isopropyl alcohol is removed overhead from the crude EMPE batch wise by simple distillation in two stages: the first at atmospheric pressure, and the second under reduced pressure. The stripped EMPE in the underflow is directed to a stripped EMPE in-process storage tank.

6a. Crude EMPE Stripping Process Vents – While filling the still and during initial distillation, the still is vented through a condenser to atmosphere via a roof mounted conservation vent set to relieve pressure at 15" WC. Final distillation occurs under vacuum while the batch still vents via condensers to 2-stage steam jets. Steam condensate from steam jet after condenser collects in a seal pot that overflows to WWT. Detailed Aspen modeling for Title V Vent Permits indicate that approximately 0.0138 lbs./day of EMPE is emitted through the atmospheric vent.

6b. Crude EMPE Stripping Distillate – Isopropyl alcohol distillate from the crude EMPE distillation is collected in a general spent solvent and filtrate storage tank for recovery and recycle. Detailed waste stream documentation indicates that no EMPE is discharged to the spent solvent storage tank.

7. Stripped EMPE In-Process Storage – Stripped, crude EMPE is transferred from the batch still to an outside, atmospheric storage tank. The stripped, crude EMPE is transferred from the storage tank to one of two batch stills for distillation of the EMPE.

7a. Stripped EMPE Storage Vessel Vent – The outside storage tank is maintained at ambient temperature and is vented via a conservation vent to the atmosphere. The conservation device relieves to atmosphere at a set pressure of 1 oz. pressure. . Detailed Aspen modeling for Title V Vent Permits indicates no EMPE emissions from the storage tanks and associated vents.

8. EMPE Distillation – Two batch stills with packed columns separate EMPE from low boilers and unconsumed reactants via vacuum distillation. Distillate collected in a first cut receiver (unconsumed reactants in the EMAA preparation process) is recycled for use in the EMAA reactor. Distillate in a second cut receiver is dropped back to the still pot for distillation with the next batch. A product cut receiver collects EMPE. Every 5th still batch, still residue (high boiling tar) is discharged via a tar dilution tank to a hazardous waste dumpster for incineration. Product receivers containing EMPE are transferred directly to an outside storage tank.

Each batch of distilled EMPE is analyzed by gas chromatography for EMPE assay and residual EMAA. The EMPE product assay averages 99.0%. The presence of EMAA is rarely detected in distilled EMPE, although there is an occasional batch with an elevated level of 0.1-0.3% EMAA. The detection limit for EMAA in the analysis is estimated to be approximately 200 ppm.

8a. EMPE Distillation Process Vents – The batch stills vent via condensers to a 2-stage steam jet system. Steam condensate from the steam jet after condenser collects in a seal pot that overflows to WWT. The tar dilution tank, condenser, and receiver vent to atmosphere via a roof vent. First cut receivers are discharged to an indoor First Cut storage tank for further distillation and recycle to normal production. The First Cut storage tank is vented to atmosphere via a roof vent and is maintained at ambient temperature. Detailed Aspen modeling for Title V Vent Permits indicates no EMPE emissions from the vacuum distillation and associated vents.

8b. EMPE Distillation Organic Wastes (Tars) – Every fifth distillation batch, tar residue in the batch still is discharged via a tar dilution tank to a hazardous waste disposal facility. Detailed waste stream documentation indicates that approximately 63 pounds per batch (19,000 pounds per year) of PM 1096 Tar is discharged to the hazardous waste disposal facility for incineration.

8s. EMPE Distillation Sampling – A 4 oz. sample is collected from a sample valve in the product distillate receiver. The operator wears safety glasses and a face shield or safety goggles, rubber gloves, a plastic apron over flame retardant coveralls, and static dissipating leather shoes.

9. EMPE In-Process Storage – Distilled EMPE is transferred to an outside storage tank where it is stored at ambient temperature and atmospheric

pressure. It is pumped from the storage tank to a reactor for conversion to CD-3 Sulfonamide.

9a. EMPE In-Process Storage Vessel Vent – The distilled EMPE outside storage tank maintains a slight nitrogen blanket and is vented to the atmosphere through a conservation vent set to relieve pressure at 1 oz. Detailed Aspen modeling for Title V Vent Permits indicates no EMPE emissions from the storage tank and associated vents.

10. CD-3 Sulfonamide Preparation – EMPE is consumed as a reactant in the preparation of CD-3 Sulfonamide by reaction with methane sulfonyl chloride. An aqueous decant from the CD-3 Sulfonamide reactor separates water containing unconsumed reactants and by-products from the product, present as an upper oil layer. Analytical testing is conducted on every batch of CD-3 Sulfonamide produced. The data indicate that EMPE is usually undetected in the CD-3 Sulfonamide product, and occasionally detected at less than 0.1 area percent by gc. CD-3 Sulfonamide product is transferred to an outside storage tank and is maintained at approximately 60ºC.

Each CD-3 Sulfonamide batch is analyzed by gas chromatography for CD-3 Sulfonamide assay, residual EMPE, and residual EMAA. The CD-3 Sulfonamide product assay average is 99.2%. The average EMPE content is 0.03%, with occasional batches with an elevated level of 0.1-0.4%. EMAA is not detected in CD-3 Sulfonamide. The detection limit for both EMPE and EMAA in the analysis is estimated to be approximately 200 ppm.

10a. CD-3 Sulfonamide Preparation Process Vents – The CD-3 Sulfonamide reactor is vented to atmosphere via a caustic scrubber discharge to roof mounted vents. Detailed Aspen modeling for Title V Vent Permits indicate no EMPE emissions from the CD-3 Sulfonamide reactor and associated vents.

10b. CD-3 Sulfonamide Preparation Aqueous Decant – The aqueous decant is sent to the industrial sewer through conductivity probes that detect the CD-3 Sulfonamide interface. The closed industrial sewer system is directed to a hazardous waste disposal and water treatment facility (WWT). Detailed waste stream documentation indicates that approximately 67 pounds per batch (~60,000 lbs./yr.) of EMPE are discharged to the WWT via the decant steps.

10s. CD-3 Sulfonamide Preparation Sampling – A 4 oz. dip sample is collected from the CD-3 Sulfonamide reactor via the open loading port. The operator wears safety glasses and a face shield or safety goggles, rubber gloves, a plastic apron over flame retardant coveralls, and static dissipating leather shoes.

11. CD-3 Sulfonamide In-Process Storage – CD-3 Sulfonamide is stored in a outside atmospheric storage tank and maintained at approximately 60ºC. It is

transferred from the outside storage tank to a reactor for conversion to CD-3 Nitroso.

11a. CD-3 Sulfonamide In-Process Storage Vessel Vent – The CD-3 Sulfonamide outside storage tank maintains a slight nitrogen blanket and is vented to atmosphere through a conservation vent set to relieve pressure at 1 oz. Detailed Aspen modeling for Title V Vent Permits indicates no EMPE emissions from the storage tank vents.

12. CD-3 Nitroso Preparation – CD-3 Sulfonamide is converted to CD-3 Nitroso by treatment with nitrous acid in aqueous solution. The product is crystallized from aqueous isopropyl alcohol and washed multiple times with water. The water-wet solid is isolated by filtration, and transferred to an autoclave mix tank for conversion to color developer CD-3.

Neither EMPE nor EMAA has ever been detected in CD-3 Nitroso, despite extensive efforts to characterize fully the impurity profile of CD-3 Nitroso by HPLC at the ppm levels.

12a. CD-3 Nitroso Preparation Process Venting – CD-3 Sulfonamide is consumed in the CD-3 Nitroso reactor. The CD-3 Nitroso reactor is vented to the atmosphere via an open loading port. Detailed Aspen modeling for Title V Vent Permits indicates no EMPE emissions from this reactor and associated vents.

12b. CD-3 Nitroso Preparation Isopropyl Alcohol Recovery – Unconsumed reactants, contaminants, and aqueous isopropyl alcohol are decanted from the CD-3 Nitroso reactor to purify the product. This decant stream is directed to a spent solvent storage for recovery via distillation and recycle of the isopropyl alcohol. Routine analytical analysis of the distillation column aqueous underflow and isopropyl alcohol takeoff indicate no detection of EMPE. Detailed waste stream documentation indicates that no EMPE is discharged to the WWT via this decant step.

12c. CD-3 Nitroso Preparation Aqueous Washes – CD-3 Nitroso is further purified with the application of water washes to remove inorganic salts. The water washes are decanted via a closed industrial sewer for treatment in a hazardous waste disposal and water treatment facility (WWT). Detailed waste stream documentation indicates that no EMPE is discharged to the WWT via this decant step.

C. Data on “Presence in Distributed Product”

CD-3 Nitroso is subsequently converted to color developer CD-3 , which is the distributed product derived from EMAA and EMPE. This conversion comprises catalytic hydrogenation, carbon treatment, conversion to the sulfuric acid salt, crystallization, and vacuum drying. Neither EMAA nor EMPE has ever been

detected in the CD-3 distributed product by HPLC, despite extensive characterization of the impurity profile at ppm levels.

EMAA is rarely detected in distilled EMPE [8]; it is never detected in CD-3 Sulfonamide [10]. The detection limit is estimated to be 200 ppm. The average EMPE content in CD-3 Sulfonamide [10] is 0.03%. The conversion of CD-3 Sulfonamide to the final distributed product, CD-3, proceeds through a nitrosation, purification, isolation, catalytic hydrogenation, carbon treatment, isolation, and vacuum drying. If no chemical transformation should occur for EMAA and EMPE in these steps, which is highly unlikely, it would be expected that the small quantities present in the CD-3 Sulfonamide would be removed through these multiple processing steps. However, since both EMAA and EMPE are active nitrosation substrates, it is fully reasonable to conclude that each, if present, would undergo nitrosation and a change in chemical structure and identity at the CD-3 Nitroso preparation step. Consequently, it may be concluded with a high degree of confidence that neither EMAA nor EMPE are present in the distributed product.

II. Information on Transport

This section is not applicable, since the structures of interest, EMAA and EMPE, are manufactured and consumed on site.

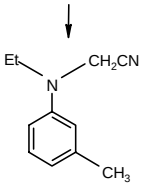
III. Supporting Evidence

Eastman does not market these two materials and is not aware of an end-use other than that of an intermediate in the previously described document. Furthermore, Eastman is believed to be the sole manufacturer of this material.

Attachment I

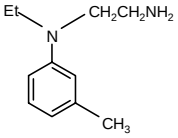
Overall Reaction Chemistry and Manufacturing Flow

REACTANTS



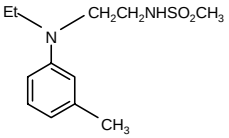
EMAA

EMAA
CAS Number 063133-74-4
Ethyl (3-methylphenyl)amino acetonitrile



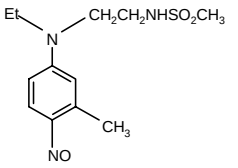
EMPE

EMPE
CAS Number 019248-13-6
N-Ethyl-N-(3-methylphenyl)-1,2-ethanediamine



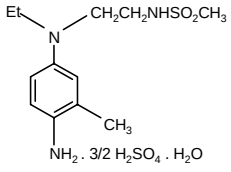
CD-3 Sulfonamide

CD-3 Sulfonamide
CAS Number 037717-68-3
N-(2-(Ethyl(3-methylphenyl)amino)ethyl)methanesulfonamide



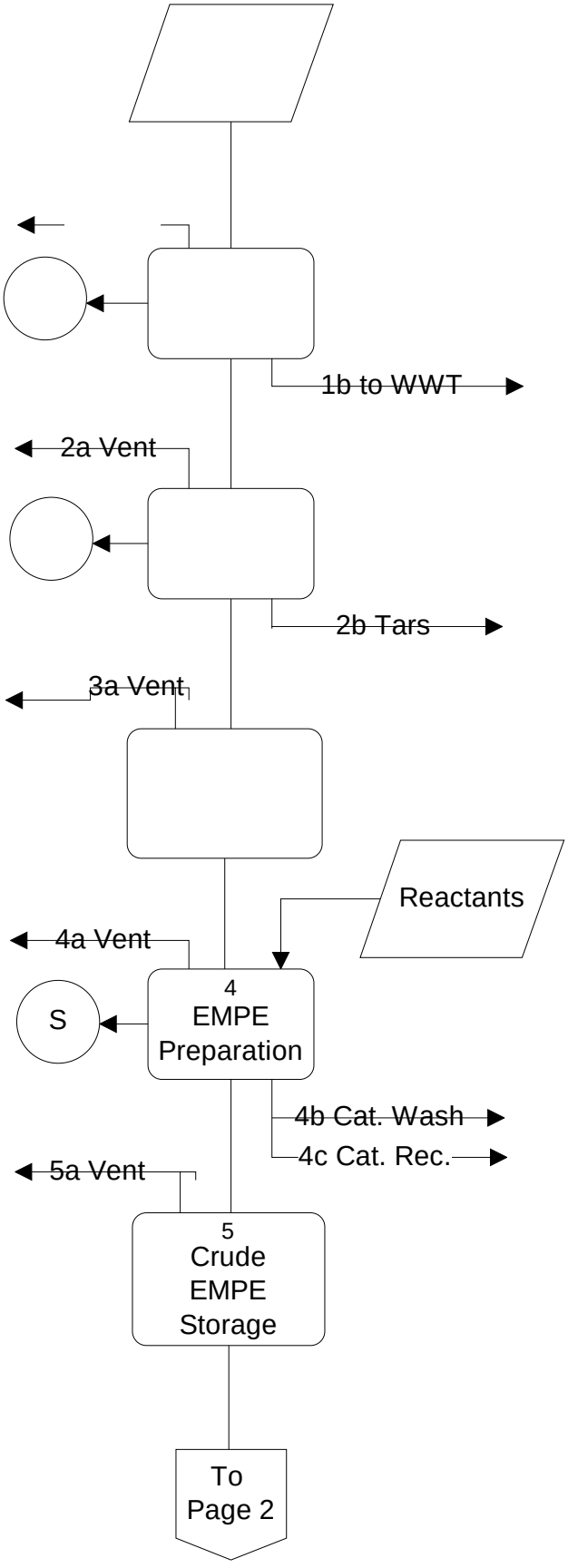
CD-3 Nitroso

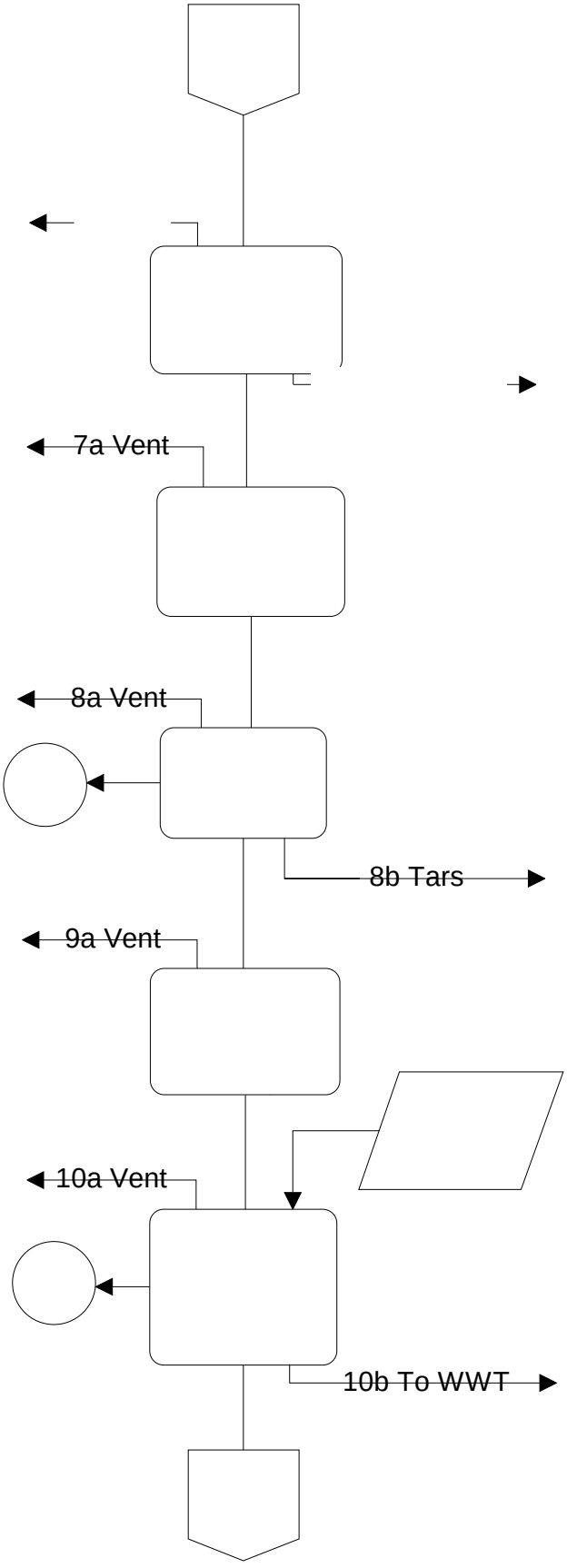
CD-3 Nitroso
CAS Number 056046-62-9
N-(2-(Ethyl(3-methyl-4-nitrosophenyl)amino)ethyl)methanesulfonamide

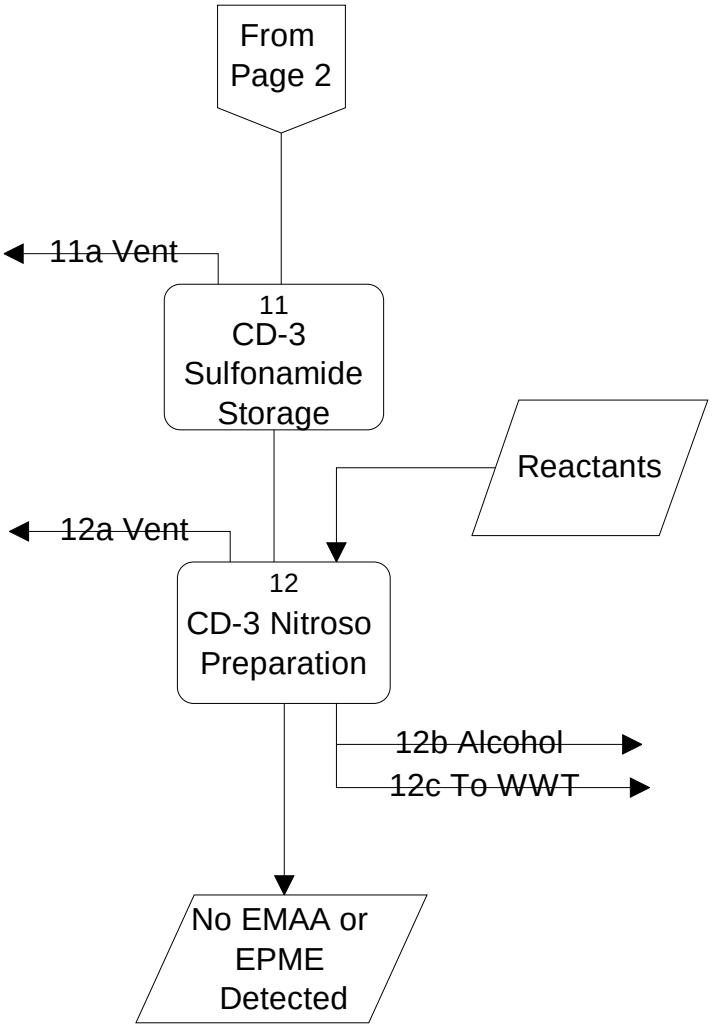


Color Developer CD-3

Color Developer CD-3
CAS Number 025646-71-3
4-(N-ethyl-N-2-methanesulfonylaminoethyl)-2-methylphenylenediamine
sesquisulfate monohydrate


1b to WWT
2a Vent
2b Tars
3a Vent
Reactants
4a Vent
S
4
EMPE
Preparation
4b Cat. Wash
4c Cat. Rec.
5a Vent
5
Crude
EMPE
Storage
To
Page 2


7a Vent
8a Vent
8b Tars
9a Vent
10a Vent
10b To WWT


From Page 2
11a Vent
11
CD-3 Sulfonamide Storage
Reactants
12a Vent
12
CD-3 Nitroso Preparation
12b Alcohol
12c To WWT
No EMAA or EPME Detected